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Potential use of cotton dust as filler in the production of thermoplastic composites

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Abstract

The effect of cotton dust as filler on the mechanical and thermal properties of polypropylene composites was investigated and the results were compared with the properties of wood plastic composites. Cotton dust was obtained from the dust filtration system located in a textile manufacturing unit. Different mixtures of cotton dust (30 to 60 wt%) or wood flour (30 to 60 wt%) were compounded with polypropylene with a coupling agent (maleic anhydride grafted polypropylene 3 wt%) in a twin-screw co-rotating extruder. The test specimens were produced by injection molding machine. The tensile strength and flexural modulus of the specimens improved with the increase in the filler content. There was no significant difference in the strength and modulus values between the cotton dust and wood flour filled composites. The highest thermal stability was found to be in the composites produced with 40 wt% of cotton dust according to the results of differential scanning calorimetry analysis. Based on the findings obtained from the present study, the optimum mechanical and thermal properties for the filled polypropylene composites were found to be a 50/50/3 formulation of cotton dust, polypropylene, and maleic anhydride grafted polypropylene, respectively.

Keywords

Cotton dust, wood flour, mechanical properties, thermal properties, thermoplastic composites

Introduction

Cotton dust is the dust of pure white cotton, which is captured by air in environment during the production of yarns with cotton. It is too short a fiber, which cannot be converted to any textile application.[1] Cotton dust generated in various sections of the yarn manufacturing process causes breathing problems to workers.[2] Cotton dust consists of 50–80 wt% fiber fragments, leaf and husk fragments, 10–25 wt% sand, and some water-soluble material.[3] The fine dust passes with the air through the perforations of the wheel. It is then discharged with air to the dust settling chamber or dust filtration system. Since cotton dust is generated as waste in large volumes by the textile industry, it needs to find ways for its sustainable recycle and reuse.

Wood plastic composites (WPCs) have been recently used in decking and siding industries due to their good biological durability high mechanical properties, and high dimensional stability.[4] Lignocellulosic materials such as wood flour and agricultural wastes (rice husk, pine cone, sunflower stalk) as filler are used in the thermoplastic composite industry.[5–9] In particular, wood flour is extensively used in the production of thermoplastic composites due to its significant advantages such as low price and density, high mechanical properties, availability, easy processing conditions, nonabrasive nature, and renewable resource. However, the sustainable utilization of wood material has been negatively affected by increasing population of the world. Cotton dust can play an important role as an alternative to wood flour in the production of filled thermoplastic composites.

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This may be one of the most efficient uses of cotton dust in high value-added materials.

There are a number of ways in which cotton dust can be disposed off sustainably such as biogas production, vermicomposting, and as a fuel for boiler. However, an extensive literature search has not revealed any information about the utilization of cotton dust as filler in the production of the thermoplastic composites. The goal of this study was to determine the mechanical and thermal properties of the polypropylene (PP) composites filled with different amounts of cotton dust and compare the results with the properties of WPC.

Experimental details

Materials

PP granulates were supplied by Likom PP Company in Ukraine. Its density and melt flow rate (230°C / 2.16 kg) were 0.90 g/cm^3 and 6.5 g/10 min, respectively. The coupling agent namely maleic anhydride modified homopolymer polypropylene (MAPP) (Optim P-425, MFI/190°C; 2.16 kg = 110 g/10 min, density: 0.91 g/cm^3) was supplied by Pluss Polymers Pvt. Ltd. in India.

Pine (*Pinus sylvestris*) wood flour having 0.25 mm size was obtained from a commercial WPC manufacturer (*Semawood* company) in Cerkezkoy, Tekirdag, Northwest Turkey. Pine wood flour without bark was dried in a laboratory oven with a fan at 100°C for 24 h to the moisture content of 0–1% before compounding process.

Cotton dust was obtained from dust filtration system located in a commercial textile manufacturer in Kahramanmaras, Turkey (Figure 1). The dust filtration system was opened by the operator and then the accumulated cotton dust on the filter was used as the raw material in the production of the thermoplastic composites. The average size of cotton dust was found to be 0.25 mm. Cotton dust was dried in an oven at 100°C for 24 h to a moisture content of 0–1% based on the oven-dry solid weight of cotton dust.

Figure 1. Cotton dust obtained from dust filtration system located in a textile manufacturer.

Preparation of thermoplastic composites

The pre-mixed filler (wood flour or cotton dust), PP, and coupling agent (MAPP) were compounded with a lab-scale twin-screw co-rotating extruder (length/dimater: 30). The barrel temperatures of the four zones in the extruder having a screw rotation of 30 r/min were set as 180°C, 185°C, 185°C, and 190°C from feeding to die zones. The compounded material was immediately cooled down in a water bath and pelletized. The pellets were then dried in an oven at 90°C for 24 h before the injection molding process. The pellets were directly injection molded into the ASTM test specimens using an injection molding machine with 60 ton capacity at a molding temperature of 190°C and 5 MPa pressure. The specimens were conditioned at a temperature of 23°C and relative humidity of 50% according to ASTM D 618.[10] The density values of the specimens varied from 1.10 to 1.15 g/cm^3. The experimental design is presented in Table 1.

Chemical analysis of wood flour and cotton dust

The wood samples were ground in a Wiley mill to obtain wood flour. It was then sieved to 40–100 mesh fractions for the chemical analysis. The cotton dust obtained from the textile mill was directly used for the chemical analysis. The ash contents of the both wood flour and cotton dust were determined according to the Tappi method T-211 om-93.[11] The extraction was performed in a Soxhlet system according to the Tappi method T-204 cm-97.[12] This method was repeated using several different solvents in succession so that the total extractive content was determined. The delignification process was applied in order to determine the chlorite holocellulose portion, and this was performed by adjusting the active chlorine content of the $NaClO_2$ solution (ca. 20%) iodometrically.[13,14]

Determination of mechanical properties of composites

The flexural tests were conducted in accordance with ASTM D 790[15] using a Lloyd testing machine at a rate of 1.3 mm/min crosshead speed. The dimensions of the test specimens were 3.5 mm × 13.2 mm × 128 mm. The tensile tests were performed according to the

Table 1. The composition of the polypropylene composites filled with wood flour or cotton dust.

Composite code (wt%)	Wood flour (wt%)	Cotton dust (wt%)	Polypropylene	Coupling agent (MAPP) (wt%)
A	30	0	70	3
B	40	0	60	3
C	50	0	50	3
D	60	0	40	3
E	0	30	70	3
F	0	40	60	3
G	0	50	50	3
H	0	60	40	3

ASTM D 638.[16] The tensile specimens were tested with a crosshead speed of 5 mm/min in accordance with ASTM D 638.[16] The izod pendulum impact bending strength of the notched specimens (notch tip radius: 0.25 mm) was performed according to ASTM D 256[17] using an impact testing machine. Seven specimens were used for each test method.

Differential scanning calorimetry analysis

Melting and crystallization behavior of the composites were studied in a heat-flux type differential scanning calorimeter (DSC) (PerkinElmer DSC 8000) according to ASTM D 3418.[18] The effect of increasing amount of the cotton dust or wood flour on the thermal properties of the composites were determined on the test samples of 5–6 mg. Each type of composite formulation was placed in an aluminum pan. Then, the sample was heated from 35 to 250°C at a heating rate of 10°C/min at a nitrogen flow rate of 20 mL/min from room temperature to 250°C.

Degree of crystallinity (X_c, %) was determined from the enthalpy of the second melting values using following equation

$$X_c = \frac{\Delta H_m}{(1-\alpha) \times \Delta H_m^{\circ}} \times 100 \quad (1)$$

where ΔH_m is the enthalpy of the second melting of the samples (J/g), ΔH_m^o is the enthalpy value of melting of a 100% crystalline form of PP (209 J/g) and $(1-\alpha)$ is the weight fraction of polymer into the composite material.

Thermogravimetric analysis

The thermogravimetric analysis (TGA) of each type of composite was carried out in an inert environment of gas nitrogen flowing 20 mL/min using a PerkinElmer STA 6000 analyzer. The specimens having a weight of 17–20 mg were heated from 50 to 600°C at a heating rate of 20°C/min. The corresponding weight loss (%) and its derivative weight loss (min/%) were recorded.

Fourier transform infrared spectroscopy

A Shimadzu IR Prestige-21 Fourier transform infrared (FTIR) spectrometer (Tokyo, Japan) equipped with attenuated total reflectance (ATR) was used to characterize cotton dust from textile mill and D-cellulose from cotton. Spectra were recorded between 600 cm^{-1} and 4000 cm^{-1}, with 16 scans per experiment and a resolution of 8 cm^{-1}. The spectra for each group were transformed into absorbance spectra, which were averaged before the baseline correction and normalization.

Statistical analysis

Analysis of variance (ANOVA) ($p < 0.05$) was used to determine the effect of amounts of cotton dust and wood flour on the selected mechanical and thermal properties of the filled thermoplastic composites. Significant differences among the average values of the composite types were determined using Duncan's multiple range tests.

Results and discussion

Mechanical properties

The mechanical properties of the composites and significant differences between the composite groups are presented in Table 2. The flexural properties of the composites decreased with increasing amount of the filler. There was no significant difference ($p < 0.05$) in the flexural strength and flexural modulus values between the cotton dust filled PP composites and wood flour filled PP composites as the amount of the filler increased from 30 to 60 wt%. The PP composites filled with the wood flour had higher flexural strength and modulus than those of the PP composites filled with the cotton dust but this was not significant. For example, the average values of the flexural strength and flexural modulus of the composites containing 50 wt% the wood flour (composite code: C) were found to be 45.4 MPa and 2818 MPa as compared to the composites containing 50 wt% cotton dust (composite code: G), which were 44.3 MPa and 2784 MPa, respectively. This was mainly due to the lower cellulose content of the cotton dust. According to Bledzki et al.[19] and Bledzki and Gassan,[20] an increase in the composite's strength can be ascribed to higher cellulose, as well as better dispersion and adhesion to the matrix. Based on the chemical analysis results, the amounts of

Table 2. Mechanical properties of the polypropylene composites filled with wood flour or cotton dust.

Composite type[1]	Mechanical properties					
	Flexural strength (MPa)	Flexural modulus (MPa)	Tensile strength (MPa)	Tensile modulus (MPa)	Total elongation at fracture (%)	Impact strength (notched) (J/m)
A	50.4 (3.3) a	2184 (605) a	23.8 (2.5) a	1169 (111) ab	5.4 (1.67) a	32.9 (2.8) a
B	45.2 (4.9) bc	2534 (303) ab	22.4 (2.2) ab	1296 (63) bc	4.1 (0.62) b	31.2 (2.0) a
C	45.4 (8.4) bc	2818 (238) abc	19.9 (1.9) bcd	1355 (96) c	3.7 (0.49) b	30.0 (1.3) ab
D	44.1 (7.5.) b	3470 (636) c	17.3 (0.9) de	1569 (148) d	1.8 (0.29) c	27.3 (3.4) b
E	48.4 (7.7) ac	2136 (282) a	23.0 (4.2) a	1078 (64) ab	5.4 (0.87) a	31.6 (1.5) a
F	46.1 (7.2) bc	2527 (326) ab	21.3 (1.1) abc	1182 (60) a	4.0 (0.37) b	30.2 (0.8) ab
G	44.3 (2.4) b	2784 (589) ab	18.9 (1.1) cde	1264 (41) bc	3.6 (0.50) b	27.5 (3.2) b
H	42.8 (6.9) b	3197 (652) bc	16.4 (1.3) e	1494 (102) d	1.4 (0.12) c	23.1 (1.6) c

The values in the parentheses are standard deviations. Groups with same letters in column indicate that there is no statistical difference ($p < 0.05$) between the specimens according to Duncan's multiple range test.

holocellulose, extractives, and ash of the wood flour were found to be 89.67%, 5.93%, and 4.56% while these contents of the cotton dust were found to be 72.11%, 5.53%, and 0.40%, respectively. The better interfacial adhesion between the wood flour and PP, due to the high cellulose content, increases the toughness or ductility.[7]

The decreases in the mechanical properties of the PP composites containing increased amount of the cotton dust can be attributed to considerably lower holocellulose content in the cotton dust than in the wood. Similar results were observed in the previous studies.[5–8] For example in the literature, flexural strength and flexural modulus values were found to be 55.8 MPa and 4555 MPa for polymer composites produced with 40% beech wood flour and 57% PP with 3 wt% MAPP while these properties were found to be 40.4 MPa and 3244 MPa for the thermoplastic composites produced with 40 wt% olive mill sludge and 57 wt% PP with 3 wt% coupling agent, respectively.[6] Bajwa et al.[21] was found to be 1017.8 N/mm^2 and 11.8 N/mm^2 for the flexural modulus and flexural strength of the thermoplastic composites prepared from 50 wt% flour of cotton burr and sticks, 40 wt% HDPE composites, 6 wt% lubricant, and 4 wt% mineral filler.

The plastic lumber standard ASTM D 6662[22] was used for comparing the results of the flexural properties of the specimens. The minimum requirements for the flexural modulus and flexural strength of polyolefin-based plastic lumbers specified by ASTM 6662 as 340 N/mm^2 and 6.9 N/mm^2, respectively. The WPCs produced with the cotton dust had flexural strength (48.4–42.8 N/mm^2) and flexural modulus (2136–3197 N/mm^2) values that were well over the requirements by the standard.

The tensile properties of the composites showed a similar trend to the flexural properties. As the amount of the cotton dust increased from 30 to 60 wt%, tensile strength of the PP composites filled with the cotton dust or wood flour did not show significant differences. A similar result was found in the PP composites filled with cotton dust or wood flour, except for the tensile modulus at 40 wt% filler content (Table 2). As the amount of the cotton dust increased from 30 to 60 wt%, the tensile strength of the PP composites decreased from 23.0 to 16.4 MPa while the flexural modulus increased from 1078 to 1494 MPa. A similar trend was observed for the wood flour filled PP composites. The decreases in the tensile strength and flexural strength of the PP composites filled with the wood flour or cotton dust might be due to the dissimilarities and lack of the adhesion between the nonpolar PP matrix and polar filler. Similar results were also reported in the previous studies.[5–8] It is worthy of note that the tensile and flexural modulus of the PP composites progressively increased with the addition of cotton dust or wood flour.

The tensile strength of the composites containing the wood flour was significantly higher than that in the composites containing the cotton dust. This could be explained by a strong interfacial adhesion between the PP and wood flour due to their higher cellulose content, since cellulose is the main component providing the wood's strength and structural stability.[7] The FTIR-ATR spectrum of the cotton dust from the textile mill and D-cellulose from cotton is presented in Figure 2. The both spectrum had similar peaks at the characteristic wavenumber of cellulose. The wavenumber characterization for D-cellulose from cotton and cotton dust from textile mill in the fingerprint area

is given in Table 3. The FTIR results indicated that the major part of the cotton dust consisted of the cotton cellulose. Based on the FTIR-ATR analysis result, it was observed that most of the holocellulose was composed of the cellulose (Figure 2). Remaining part of the cotton dust was composed of other extractives, organic, and inorganic materials.

The impact strength of the PP composites decreased with increasing the cotton dust or wood flour content (Table 2). There was no significant difference in the impact strength values between the wood filled PP composites and cotton dust filled PP composites, except for the 60 wt% filler content. For example, the impact strength of the specimens containing 40 wt% wood flour was found to be 31.2 J/m while it was found to be 30.2 J/m for the specimens containing 40 wt% cotton dust. The impact strength of the PP composites decreased from 31.6 to 23.1 J/m as the cotton dust content increased from 0 to 40 wt% while it decreased from 32.9 to 27.3 J/m for the wood flour filled PP composites. This could be due to the fact that the incorporation of cotton dust into the PP composite created more regions of stress concentration that required less energy to initiate a crack in the composite, thereby decreasing the impact strength. The decrement in the impact strength of the composites containing a higher amount of the filler showed that the filler reduced the polymer chain mobility and therefore its ability to absorb energy during the fracture propagation. The poor interfacial bonding between filler and thermoplastics causes microcracks to occur at the point of impact, which results in the cracks to easily propagate in the composite.[8] The values of the total elongation at fracture significantly decreased with increasing amount of the filler content. The values of the cotton dust filled PP composites were slightly lower than the values of the wood flour filled PP composites. There was no significant difference in the total elongation values between the wood flour and cotton dust filled PP composites (Table 2).

Thermal analysis

DSC analysis. The DSC results of the PP composites filled with the cotton dust and wood flour are given in Table 4. In data from the DSC analysis, there were unremarkable changes in the first and second melting peak temperatures of the PP composites filled with the wood flour or cotton dust. The peak temperature of

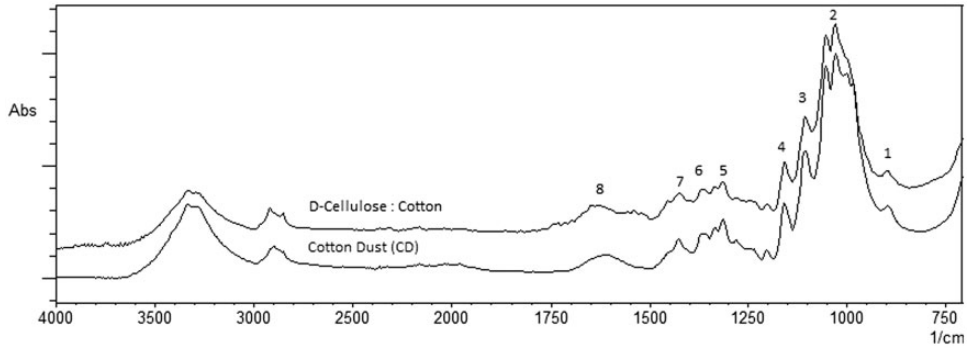

Figure 2. FTIR-ATR spectrum of cotton dust and D-cellulose from cotton.

Table 3. Wavenumber characterization for D-cellulose from cotton and cotton dust in the fingerprint area.

Wavenumber (cm^{-1})	Compounds	Peak number from Figure 2
897 (910–885)	C–H deformation in cellulose	1
1029 (1070–1040)	C–O in cellulose and hemicelluloses	2
1106 (1120–1075)	O–H in cellulose and hemicelluloses	3
1160 (1170–1142)	C–O–C vibration in cellulose and hemicelluloses	4
1316 (1324–1308)	C–H vibration in cellulose	5
1370 (1385–1355)	C–H deformation in cellulose and hemicelluloses	6
1426	Scissor oscillation of CH_2 groups characteristic for rotational isomers in cellulose	7
1645	Symmetric deformational oscillation of water molecules absorbed on the cellulose)	8

Table 4. The results of DSC analysis.

Composite type[a]	$T_{m,I}$ (°C)[b]	$T_{c,O}$ (°C)[c]	$T_{c,P}$ (°C)[d]	ΔH_C (J/g)[e]	$T_{m,II}$ (°C)[f]	ΔH_{II} (J/g)[g]	X_C (%)[h]
A	163.9	125.9	121.7	55.4	164.7	28.7	19.6
B	165.1	125.3	121.4	45.6	164.1	22.0	17.5
C	164.3	125.2	121.5	49.4	163.8	24.1	23.1
D	164.5	125.9	122.2	32.8	164.2	15.4	18.4
E	165.6	125.7	121.3	59.8	164.2	28.9	19.8
F	165.3	127.1	122.9	44.2	164.6	21.6	17.2
G	164.8	127.1	122.7	49.5	165.0	21.9	21.0
H	162.9	128.1	124.2	31.3	164.2	12.2	14.6

[a]Composite type.
[b]Peak temperature of first melting.
[c]Onset temperature of crystallization.
[d]Peak temperature of crystallization.
[e]Enthalpy of crystallization.
[f]Peak temperature of second melting.
[g]Enthalpy of second melting.
[h]Degree of crystallization calculated with the enthalpy of second melting.

the second melting of the PP composites filled with the cotton dust was slightly higher than that of the PP composites filled with wood flour, except for the 30 wt% filler content (Figure 3). The second melting peak temperature of the composites slightly increased with increasing the cotton dust content. However, this was not observed for the wood flour filled composites. There was a very slight difference (164–165°C) in the peak temperature of the second melting of the PP composites filled with the cotton dust between the 30 wt% and 60 wt% filler contents (Figure 3). The highest thermal stability were found to be in the composites produced with the 40 wt% the cotton dust based on the results of the peak temperature of second melting.

The enthalpy of crystallization (ΔH_c) of the PP composites filled with the cotton dust was found to be higher than that of the PP composites filled with the wood flour at 30 wt% filler content, However, this was not observed in the PP composites having a higher amount of the cotton dust (Table 3). The enthalpy of the second melting (ΔH_{II}) values decreased with increased filler content in the polymer composites. Similar values were observed for the values of the enthalpy of crystallization. The wood flour filled PP composites had higher crystallinity degree (X_c) values than the cotton dust filled PP composites, except for the 30 wt% filler content. For example, X_c of the PP composites filled with the 50 wt% wood flour was found to be 23.1% while it was found to be 21.0% for the PP composites filled with the cotton dust. The difference in the X_c values between the wood flour or cotton dust filled PP composites increased with increasing the filler content. The X_c values of the PP composites increased with increasing wood flour or cotton dust content up to 50 wt%, which could be due to the nucleation effect of wood flour.[23,24] However, further increment in the wood flour or cotton dust considerably decreased the X_c values.

TGA analysis. The TGA and derivative TGA of the PP composites filled with the cotton dust or wood flour are presented in Figures 4 and 5, respectively. The thermal degradation started at 220°C and continued up to about 480°C with two successive peaks. Two main decomposition peaks were shown with arrows on the derivative TGA thermograph (Figure 4). The first peak was about 350–375°C, which was assigned to the thermal decomposition to the cellulose and lignin while the second peak was 460–480°C was assigned to the PP (Figure 4). The first degradation was observed to be around 220°C, which was due to the hemicelluloses that were the most thermally sensitive components in the lignocellulosic filler, followed by cellulose/lignin and then PP. As for the cotton dust filled PP composites, first peak of about 350°C was mainly assigned to the degradation of cotton dust. Since the most of holocellulose of the cotton dust was composed of cellulose, the weight loss of the PP composites filled with the cotton dust was lower than that of the PP composites filled with the wood flour at a low filler content (30 wt%). However, at a high content of the filler (60 wt%), the weight loss of the PP composites filled the cotton dust was more pronounced than the PP composites filled with the wood flour (Figure 5).

As shown in Figure 4, the thermal stability of the PP composites filled with the cotton dust was generally better than the PP composites filled with the wood flour, but it was not significant. This was because the

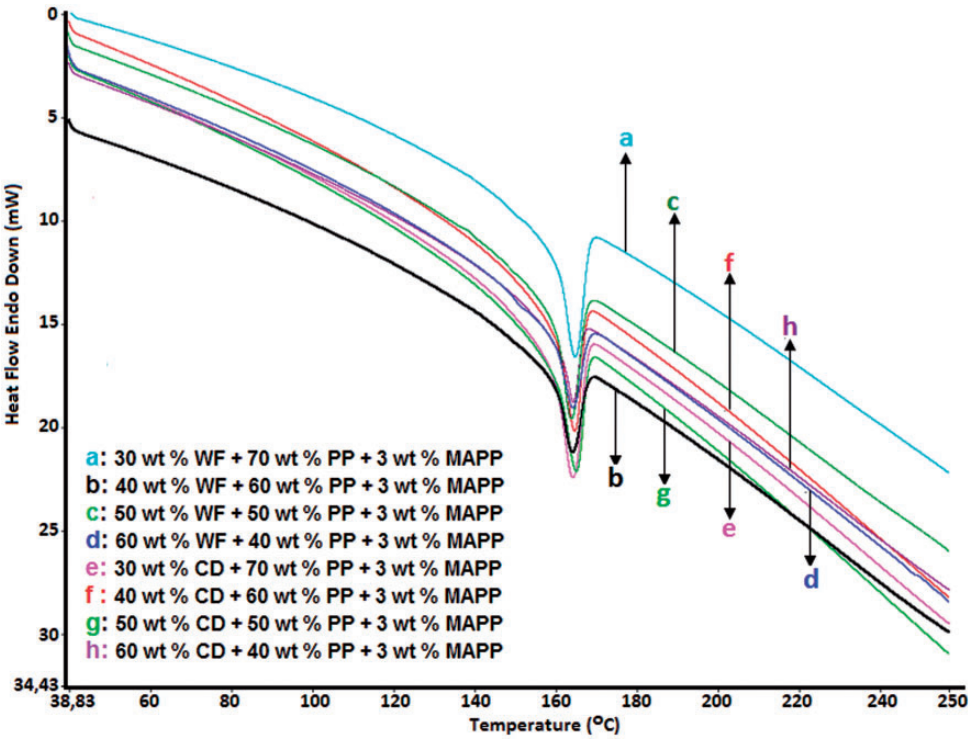

Figure 3. Heat flow endo down curves of the thermoplastic composites.
WF: wood flour; PP: polypropylene; MAPP: maleic anhydride grafted PP; CD: cotton dust.

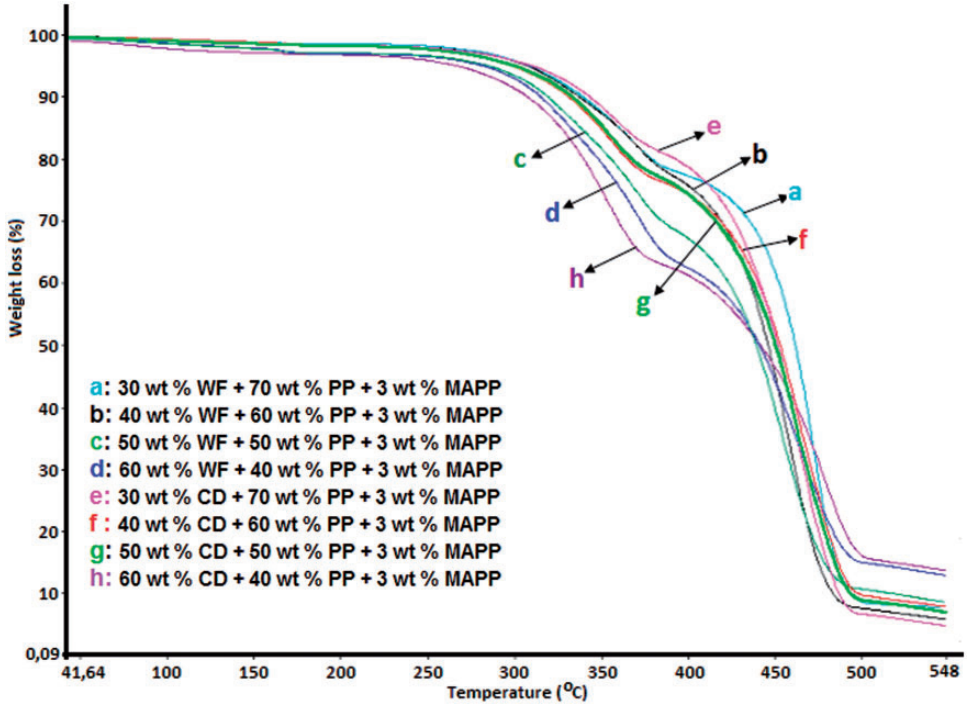

Figure 4. TGA weight loss curves of the thermoplastic composites.
WF: wood flour; PP: polypropylene; MAPP: maleic anhydride grafted PP; CD: cotton dust.

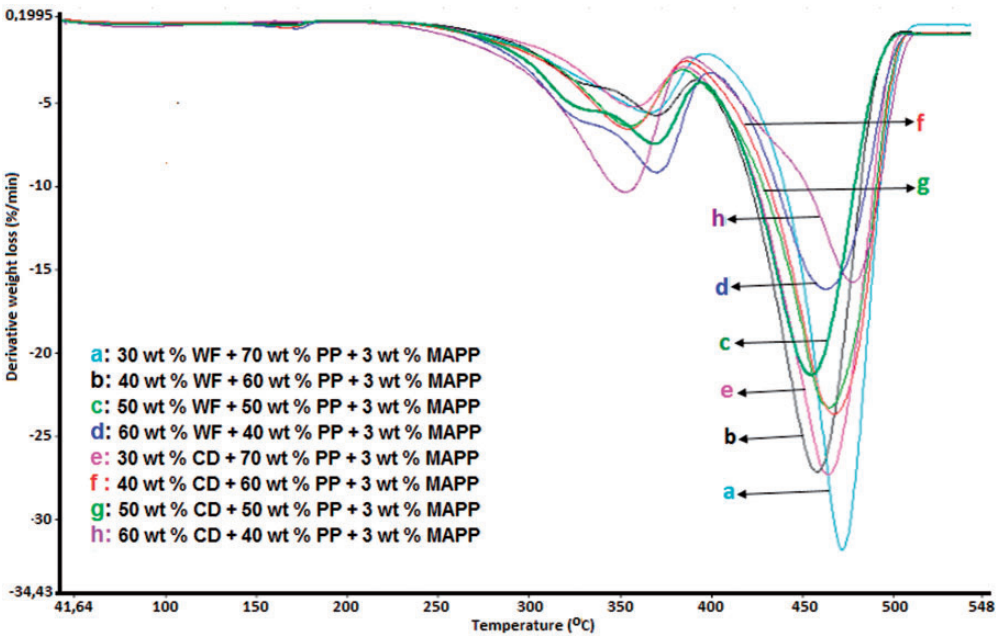

Figure 5. Derivative weight loss curves of the thermoplastic composites composites.
WF: wood flour; PP: polypropylene; MAPP: maleic anhydride grafted PP; CD: cotton dust.

degradation temperatures of the PP composites filled with the cotton dust were higher than those of the PP composited filled with the wood flour wood flour (Figure 4). The cotton dust mainly contains the cellulose while the wood flour contains hemicellulose/cellulose. The hemicellulose made the wood had a lower thermal stability than the cotton dust based on the DSC and TGA results. Moreover, the increase in the polymer composite residues after 500°C was evident as filler content increased for the PP composites. As shown in Figure 4, the residues of the PP composites filled with cotton dust was higher than the PP composites filled with the wood flour.

In nature, the thermal degradation temperature of wood flour is lower than that of PP. The filler content with low degradation temperatures reduces thermal stability of the polymer composites, which is in good agreement with those results obtained in the previous studies.[25–28] The degradation temperatures of the filled composites were reduced by the increased amount of the cotton dust or wood flour. Furthermore, the degradation temperatures of the cotton dust or wood flour filled PP composites were much lower than that of the PP, but the degradation rate was much slower.

Conclusions

The results of the present study showed that the thermal stability and degradation temperature of the PP composites decreased with increasing mass fraction of the wood flour and cotton dust. The WPC produced with the cotton dust was more thermally stable than the WPC produced with the wood flour up to 50 wt%. The flexural and tensile modulus of the PP composites were significantly improved by the addition of cotton dust as well as wood flour. The flexural strength of the composites filled with the cotton dust or wood flour did not signficantly decreased with increasing amount of the cotton dust while the tensile strength was significantly different. The advantage of the cotton dust as a filler for the PP composites was that its mechanical properties were slightly lower than the results of the PP composites filled with the wood flour. Since most of the holocellulose of the cotton dust was composed of cellulose, the weight loss of the PP composites filled with the cotton dust was lower than that of the PP composites filled with the wood flour at a low filler content (30 wt%). The highest thermal stability was found to be in the composites produced with the 40 wt% the cotton dust according to the results of the DSC analysis. Based on the findings obtained from the present study, the optimum mechanical and thermal properties for the filled PP composites were found to be a 50/50/3 formulation of cotton dust, PP, and MAPP, respectively.

Declaration of Conflicting Interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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